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REPORT OF INVESTIGATIONS

A POLARIZING COMPARISON-MICROSCOPE FOR USE IN PETROGRAPHIC MEASUREMENTS

The work upon which this report was based was performed under a cooperative agreement between the Southern Experiment Station, Bureau of Mines, United States Department of the Interior, and the University of Alabama.

BY

GEORGE T. FAUST

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REPORT OF INVESTIGATIONS

UNITED STATES DEPARTMENT OF THE INTERIOR - BUREAU OF MINES

A Polarizing Comparison-Microscope for Use in Petrographic Measurements [1]

By George T. Faust [2]

INTRODUCTION

The petrographer engaged in examining the products resulting from the beneficiation of ores and rock minerals is constantly required to develop new means and methods of assaying microscopically the results of such operations. From time to time investigators have published descriptions of apparatus that have aided in solving problems in processes of physical separation. Thus, in 1919, Coghill and Bonardi [3] published a paper covering the application of the binocular microscope and camera lucida to the microscopic analysis of pulverized ores. Similar work has been published by Schneiderhohn [4] Schwartz [5], and Head and his associates [6], [7], [8].

The present paper goes further and considers application of the polarizing comparison-microscope to problems directly concerned with mineral dressing where it is desirable to compare analogous products, and in addition it suggests new uses in various related fields. This work was conducted under the cooperative agreements of the Southern Experiment Station of the Federal Bureau of Mines and the University of Alabama.

1/ The Bureau of Mines will welcome reprinting of this paper, provided the following footnote acknowledgment is used: "Reprinted from Bureau of Mines Report of Investigations 3503."

2/ Assistant chemist-petrographer, Nonmetals Division, Bureau of Mines, Southern Experiment Station, Tuscaloosa, Ala.

3/ Coghill, W. H., and Bonardi, J. P., Approximate Quantitative Microscopy of Powdered Ores Including the Use of the Camera Lucida: Bureau of Mines Tech. Paper 211, 1919, p. 17.

4/ Schneiderhöhn, Hans, Anleitung zur mikroskopischen Bestimmung und Untersuchung von Erzen und Aufbereitungsproducten besonders in auffallenden Licht: Gesel. deut. Metallhüten u. Bergleute, e. V., Berlin, 1922, 292 pp.

5/ Schwartz, G. M., Review of the Application of Microscopic Study to Metallurgical Problems: Econ. Geol., vol. 33, 1938, pp. 440-453.

6/ Head, R. E., The Technique of Preparing Thin Sections of Rocks: Utah Eng. Exp. Sta., University of Utah, Tech. Paper 8, 1929, p. 20.

7/ Head, R. E., Quantitative Microscopic Determination of Chalcopyrite Chalcocite, Bornite, and Pyrite in a Porphyry Ore: Bureau of Mines Rept. of Investigations 2256, 1929, 5 pp.

8/ Head, R. E., and Crawford, A. L., A Staining Method for Distinguishing Cerussite and Anglesite in Ores, Concentrates, and Tailings: Bureau of Mines Rept. of Investigations 2932, 1929, 3 pp.

Acknowledgments

The author wishes to acknowledge the encouragement and practical suggestions given by Will H. Coghill, supervising engineer of the Southern Experiment Station, Bureau of Mines. He is also indebted to R. G. O'Meara, of the same institution, for his interest in this work. O. F. Soetbeer and E. Keller of E. Leitz Co., New York City, kindly cooperated in obtaining the polarizing attachments and effecting the changes requested by the author. The author is also indebted to his colleagues and to T. N. McVay for the use of the specimens here illustrated. E. E. Creitz and G. D. Coe kindly assisted in the preparation of the prints.

COMPARISON MICROSCOPES

Comparison microscopes are in use in general microscopic work, but, since ordinary (unpolarized) light is employed their application has been limited. The modification herein described extends their employment to include measurements with polarized light and thus enhances their usefulness.

Comparison microscopes 9/ are of two types, namely, that consisting of two independent microscopes united by a common eyepiece, and the type consisting of a single microscope stand with two tubes. The latter form is suitable for microscopic-petrographic observations and is illustrated in figures 1 and 2.

For microscopic-petrographic measurements, a necessary requirement is that the two microscope tubes must be capable of simultaneous actuation to the same degree. This function is necessary to permit observation of the Becke-line (central-illumination) phenomenon, which is of fundamental importance in measurements. The Becke-line phenomenon is employed in the microscopic determination of the refractive index of minerals. For example, let us consider a thin section of rock containing the grains of two different isotropic minerals adjacent to one another and possessing a vertical contact (that is, the contact is parallel to the optical axis of the microscope.) If we focus the microscope objective on the contact so that the boundary between the two grains is a distinct line, we say that the microscope is in focus. Should the microscope objective be raised from this position, a white band of light appears to move in one of the grains; with lowering of the objective, the band moves into the other grain. This white band of light which appears on movement of the microscope tube is frequently referred to as the "Becke line." The general rule states that upon raising the tube, the band of light moves into the substance of higher refractive index. For a more complete elementary explanation of the origin and use of the Becke line the reader is referred to "Mineralogy" by Kraus, Hunt, and Ramsdell.10/

9/ Chamot, E. M., and Mason, C. W., Handbook of Chemical Microscopy: Vol. 1, John Wiley & Sons, New York, 1938, pp. 27-38 and 66-67.

10/ Kraus, E. H., Hunt, W. F., and Ramsdell, L. S., Mineralogy - An Introduction to the Study of Minerals and Crystals; McGraw-Hill Book Co., New York, 3d ed., 1936, pp. 120-122.

Legend:

1. Focusing ocular
2. Focusing collar on objective
3. Condenser
4. Objectives
5. Auxiliary condensers
6. Cap nicol
7. Gypsum plate
8. Frosted and colored glass disks
9. Ring containing the polarizer

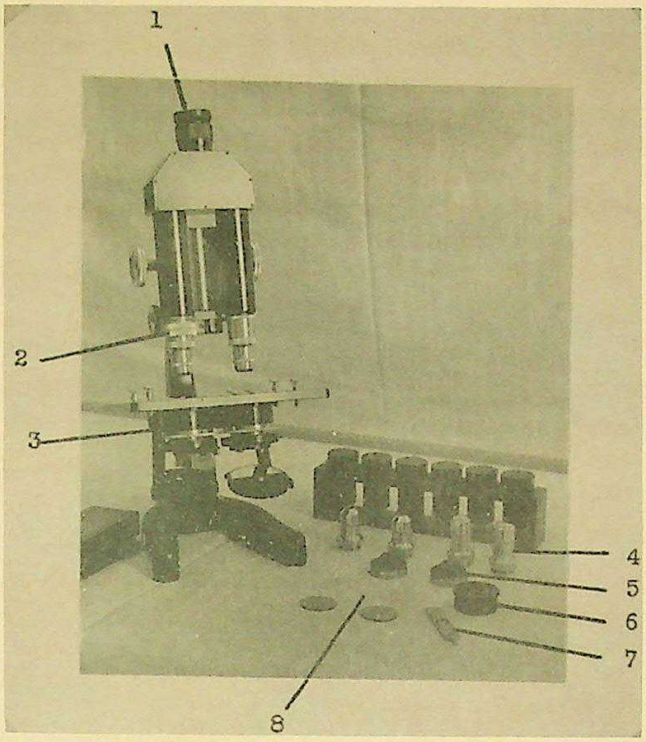

Figure 1.- Comparison microscope with accessories.

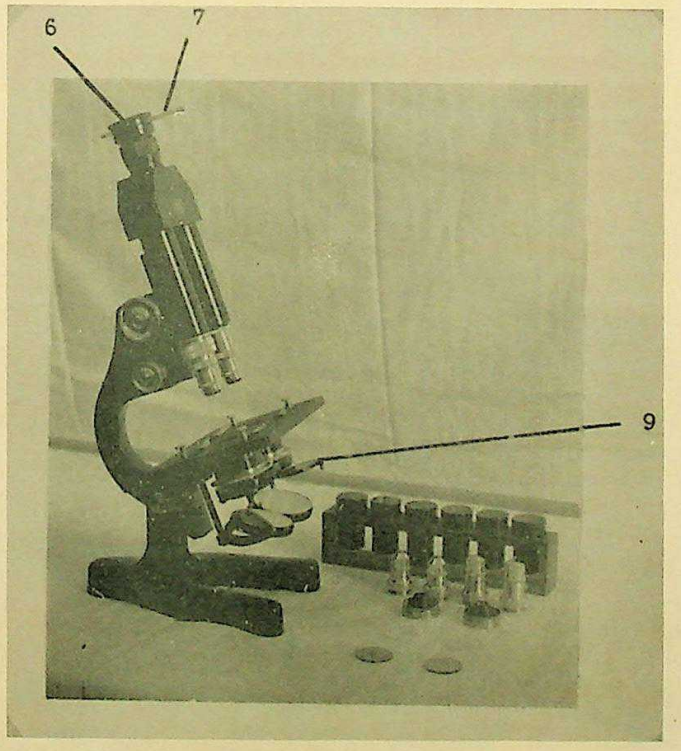

Figure 2.- Polarizing comparison microscope.

POLARIZING COMPARISON-MICROSCOPE

A decided advantage of the polarizing comparison-microscope is its ability to permit direct comparison of two quite similar specimens, allowing immediate detection of very slight and otherwise unnoticeable differences in character. Thus, the older and annoying practice of forming a mental picture of one slide on the stage of the petrographic microscope and then attempting to compare this mental image with the actual image of a second slide is entirely eliminated.

Indeed, when photomicrographs are required it is merely necessary to take one picture, in which each of the compared preparations will have identical optical conditions. The amount of time saved when many pictures are to be taken is considerable.

The microscope consists of two tubes optically united by means of prisms, a Ramsden ocular, and a focusing collar. Mechanically, the tubes are united to the same rack and are actuated by the same coarse and fine adjustments. The objectives are paired. To compensate for any slight difference in the magnifying properties of the objectives, a micrometer screw-collar is placed on one of the tubes. The stage contains two circular openings, each with its own set of slide clips. The substage apparatus consists of two separate condensers of two lenses each, having a rated numerical aperture of 1.20. Immediately beneath each of the condensers is an iris diaphragm and a supporting ring to accommodate either a blue filter glass or a plate of frosted glass. In addition, each tube has its own movable mirror.

The changes made by the author consist of the addition of a polaroid plate mounted in the ring beneath the condenser, which serves as the polarizing element, and of a special cap nicol of polaroid fitted to the Ramsden ocular. Further, a slit was made in the cap analyzer to permit insertion of a first-order gypsum test plate (See fig. 2.) These changes were carried out by the E. Leitz Co. at the author's suggestion. With this equipment, the microscope is adapted to comparison microscopic-petrographic examinations.

The value of this modified instrument lies in the fact that in many instances it permits rapid identification of a given species in a multicomponent mixture. This is accomplished by the recognition in polarized light of some one of the following diagnostic characteristics of a species - isotropism, anisotropism, order of birefringence, twinning, pleochroism, or anomalous double refraction. Species may also be distinguished by virtue of the Becke-line (central-illumination) phenomena.

Insertion of the gypsum plate in the cap nicol tends to emphasize some of the aforementioned properties and has been found especially useful in the examination of fragments having low birefringence. At times it is expedient to combine the optical characteristics mentioned above with the form of the grains, the presence of inclusions, or the presence of natural or artificial staining to permit rapid identification.

To illustrate the use of the polarizing comparison-microscope, a number of photomicrographs were taken and are shown here. In figure 3 we have samples of the same materials, garnet and quartz, viewed under polarized light with and without crossed nicols. This picture was obtained by merely sliding one of the rings containing polaroid out of the path of the light. This operation corresponds, respectively, to the insertion of and removal of the analyzer. Here we note that the garnet, being isotropic, disappears under crossed nicols. This makes it possible to estimate the percentage of quartz and garnet by examining the preparation, first with the cap nicol (analyzer) off and next with it on the ocular.

Application

The application of the polarizing comparison-microscope is divided into that in economic petrography and that in petrography.

A. Economic Petrography.

This term is suggested for describing the broad scope of application of microscopic-petrographic methods to mineral products in the following fields: (1) Mineral dressing products; (2) examination of ceramic materials, including cements; (3) comparison of constituents of pharmaceuticals, chemicals, industrial chemicals, mine and industrial dusts; and (4) selection of materials for engineering and mining. The term "economic petrography" thus stands in the same relation to petrography as does economic geology to geology.

1. Mineral Dressing - (a) In examining products from the various methods of mineral beneficiation of the nonmetallic rocks by such methods as jigging, tabling, flotation, and heavy-liquid separation, it is desirable to compare the products of successive tests to assess the quality of the operations.

Heavy-liquid separation of crushed-rock minerals into "sink" and "float" fractions has been used for some time in mineral-dressing practice. A microscopic study of various fractions to determine whether or not separations have been complete and whether or not the grains are free of associated minerals is desirable. In figures 4 and 5 we are able to examine the results obtained in the study of a kyanite schist. This crushed rock was first separated on acetylene tetrabromide (sp. gr. = 2.94). The "sink" then was passed over a magnetic separator, and the magnetic portion was separated into "sink" and "float" fractions on methylene iodide (sp. gr. = 3.31). In figure 4 the quartz "float" on 2.94 and the biotite "float" on 3.31 are compared. In figure 5 kyanite (the nonmagnetic "sink" on 2.94) and garnet, with some sulfides (the magnetic "sink" on 3.31), are compared. Only a glance through the polarizing comparison-microscope is required to answer the question: Has a good separation been obtained? The photomicrographs confirm this statement.

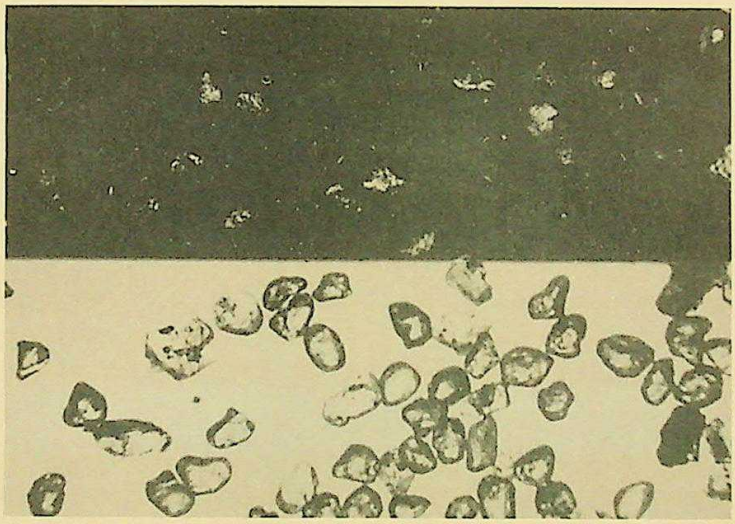

Figure 3.- A mixture of garnet and quartz sand grains viewed in the upper portion under crossed nicols, in the lower portion in plane polarized light. The garnet does not appear above owing to its isotropic character. Some of the garnets show dodecahedral form. (Approximate mag. 26X.)

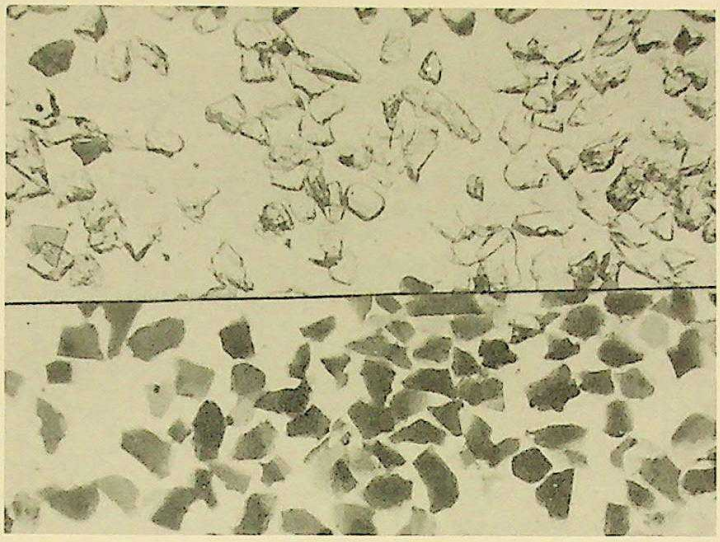

Figure 4.- Fractions of biotite and quartz separated by heavy liquids. In the biotite fraction (below) a rare grain of kyanite is found locked to biotite. The quartz fraction contains an occasional flake of biotite. (Approximate mag. 26X.)

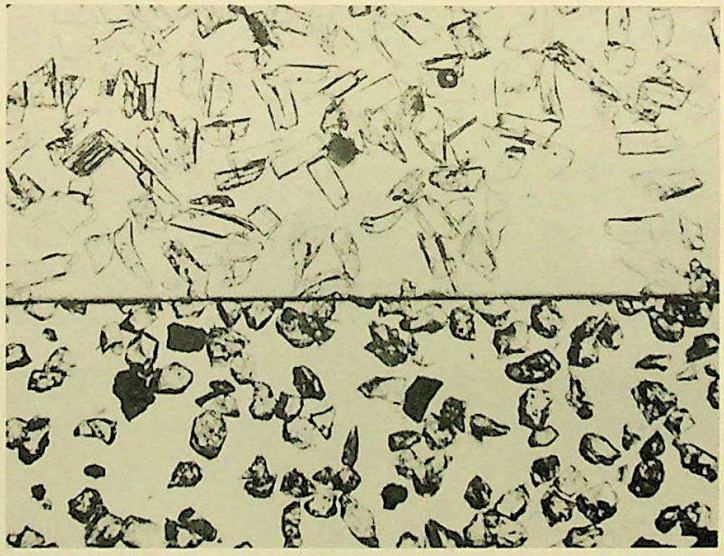

Figure 5.- Fractions of kyanite and garnet with sulfide. The kyanite concentrate (above) contains a rare flake of biotite and grain of garnet. The garnet and sulfide fraction contains a few grains of monazite and quartz. (Approximate mag. 26X.)

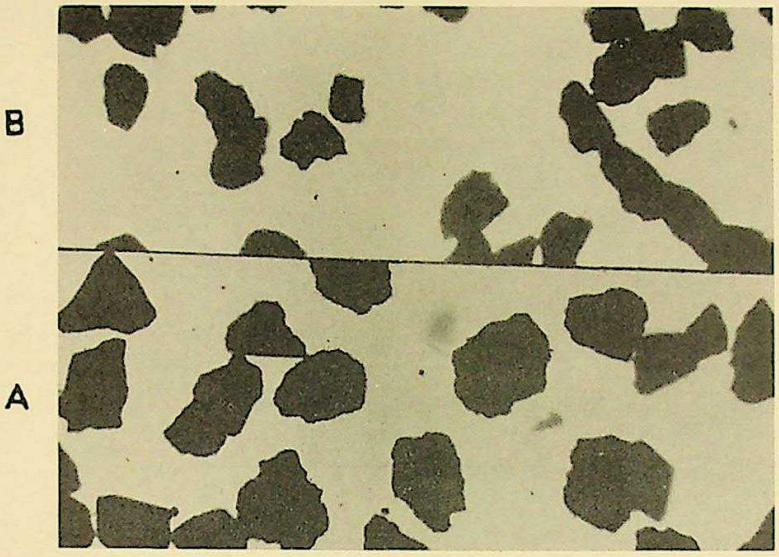

Figure 6.- Coal, wet-screened; A, Minus 150 mesh plus 200 mesh (below); B, minus 200 plus 270 mesh (above). (Approximate mag. 81X.)

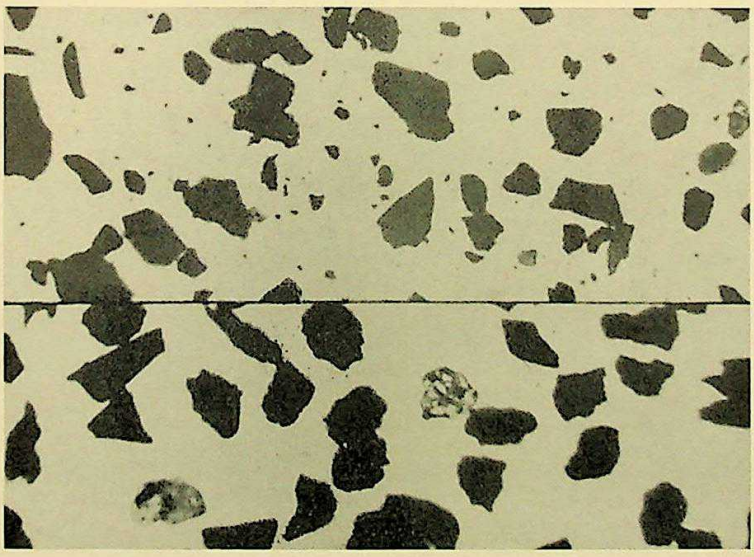

Figure 7.- Comparison of coal, wet-screened to minus 200 plus 270 mesh (below) with coal sized by sedimentation, nominal size minus 200 plus 270 mesh (above). Note occasional rock particles. (Approximate mag. 81X.)

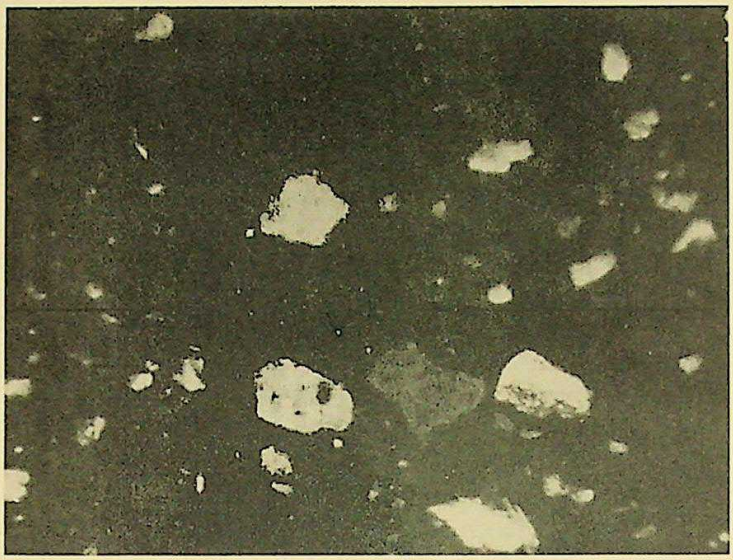

Figure 8.- Photomicrograph of two earthenware low-fired bodies containing fragments of quartz. The samples are representative areas and demonstrate readily the relative difference in quartz content. (Approximate mag., 81X. Crossed nicols.)

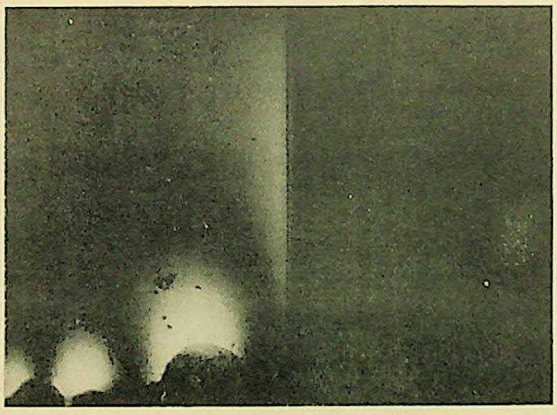

Figure 9.- Comparison of a strained and unstrained slab of glass. The slab of glass (1 inch by 1 inch by 3/16 inch) on the left is held in the jaws of a simple vise. The distribution of the strains near one of the jaws is clearly demonstrated. The slab on the right is simply lying on the stage of the microscope and has no visible indication of strain. (Approximate mag., 26X. Crossed nicols.)

The author has employed the polarizing comparison-microscope in examining the products of beneficiation of the following types of complex rocks: Kyanite-garnet-quartz-sulfide; red iron ore-clay-sand; sillimanite-quartz-biotite; spodumene-quartz-feldspar; sillimanite-kyanite-quartz; feldspar-quartz-mica; clay-quartz-mica; fluorite-calcite-quartz; graphite-sericite-quartz; phosphate-clay-sand; coal-bone.

(b) Studies of sized materials are of fundamental importance in mineral dressing.[11/] Mineral particles are separated into discrete size fractions by various means, such as screening, sedimentation, and air or liquid elutriation. To test the efficacy of such separations, the particles of successive or corresponding fractions are studied by means of the comparison-microscope. The completeness of separation on two successive screens is shown in figure 6, which is a comparison study of coal wet-screened through 150 mesh and on 200 and 270 mesh. The microscopic proof of the good quality of the separation is apparent. The separation is clean. Figure 7 is a comparison of wet-screened coal and water-sedimented coal. These are not to be taken as examples of the quality of results obtainable by wet-sedimentation methods, but merely as suitable illustrations of the usefulness of the instrument involved. The photomicrographs show unequivocally the superiority of the screened product. With crushed coal and other materials, it is not uncommon to find extraneous minerals and rock fragments. By means of polarized light it is possible to detect and thus allow for such material. Such fragments are visible in figure 7.

2. Ceramics, including cements - The polarizing comparison-microscope can be very helpful in the examination of ceramic materials. Some uses are indicated here; others will suggest themselves to workers in this field.

(a) In the study of refractories, porcelain bodies, terra cotta, and other ceramic materials, it is of value to be able to compare such properties and characteristics as the relative amounts of grog, bond development, glassy material, crystal growth, and corrosion. This is done easily with the polarizing comparison-microscope at magnifications ranging from as low as 18 diameters (3X-objective) to those produced by oil-immersion systems. Paired objectives are available throughout the range. Further, one may estimate the relative proportion of constituents of two individual samples by simple areal inspection. In figure 8 we can observe simultaneously the relative amount of coarse quartz present in two similar types of earthenware bodies. The quartz is conspicuous by virtue of its double refraction.

(b) The relative residual strain in glass objects may be compared. In figure 9 we are clearly able to recognize the presence and distribution of the strain in one of the fields. The strain was produced by placing a glass block in a simple press and exerting enough force to produce a visual strain. The photomicrograph fails to bring out the beautiful color effects produced. Prepared glass objects may be compared directly with well-annealed

11/ See Gaudin, A. M., Principles of Mineral Dressing: McGraw-Hill Book Co., New York City, 1939, pp. 51-91.

specimens or with those having the maximum amount of permissible strain. In this way, by comparison with a satisfactory piece as a standard, unsatisfactory pieces may be eliminated. If desirable, a graduated compensator could be constructed to make quantitative measurements. It would be inserted in the slot in the cap nicol.

(c) The purity of ceramic raw materials may be compared with that of standards. Likewise, one may simultaneously compare frits and ceramic mixes for homogeneity.

3. Comparison of constituents of pharmaceuticals, chemicals, industrial chemicals, mine and industrial dusts. - Qualitative comparison-microscope studies can be made on these materials to detect and compare constituents or impurities, adulterants, or fillers present. Qualitative comparisons involving percentage composition, crystal shape, particle shape, and particle size are easily made.

4. Selection of materials for engineering and mining. - In the selection of rock materials for construction purposes, it is often necessary to have comparative microscopic data on a number of specimens to determine which is most suitable. These examinations include, among other things, knowledge of the components, texture, cementing material, presence of voids, alteration, opening between grains, particle shape, inclusions, and microstructural features. The instrument herein described yields such comparative data by mere inspection.

B. Petrography.

Petrographers, in their examination of suites of specimens collected in a given area, are often called upon to make comparative studies. Two illustrations are given to show the quality of results to be expected.

1. Comparison of character and degree of alteration. - In figure 10 we can see the variation in amount of alteration present in two specimens of the same pegmatite mass taken at two different places. The example illustrated is necessarily greatly contrasted to emphasize the difference in a photomicrograph.

2. Detrital sediments. - By means of the polarizing comparison-microscopes it is possible to compare shapes, changes in mineral assemblages, and proportions of constituents in a series of samples of detritals. For illustration, the heavy minerals in a dune sand and a sand taken from a canal cut in southern Alabama are compared in figure 11, and the differences are at once apparent.

PHOTOMICROGRAPHY WITH THE POLARIZING COMPARISON-MICROSCOPE

As this comparison microscope contains two separate mirrors and condensing systems with substage diaphragms, the preparation of balanced photomicrographs of greatly contrasted materials requires care. If it is

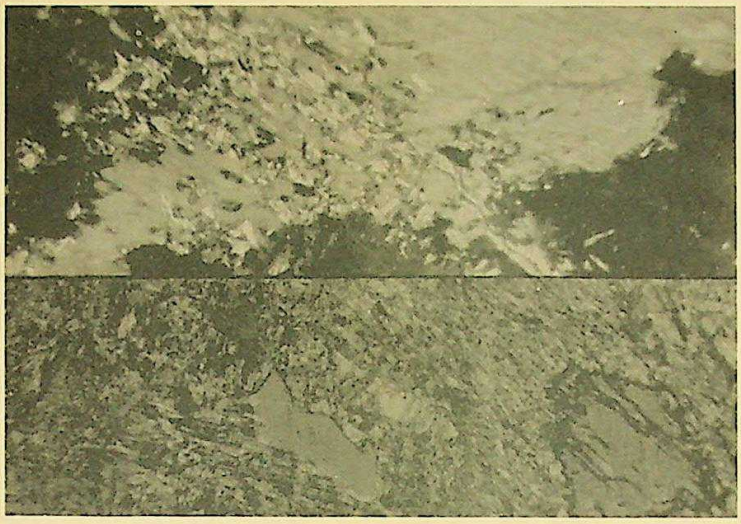

Figure 10.- Photomicrograph of two thin sections of rock specimens belonging to the same pegmatic body but from different positions within the mass. The relative amount of alteration is apparent. (Approximate mag., 81X. Crossed nicols.)

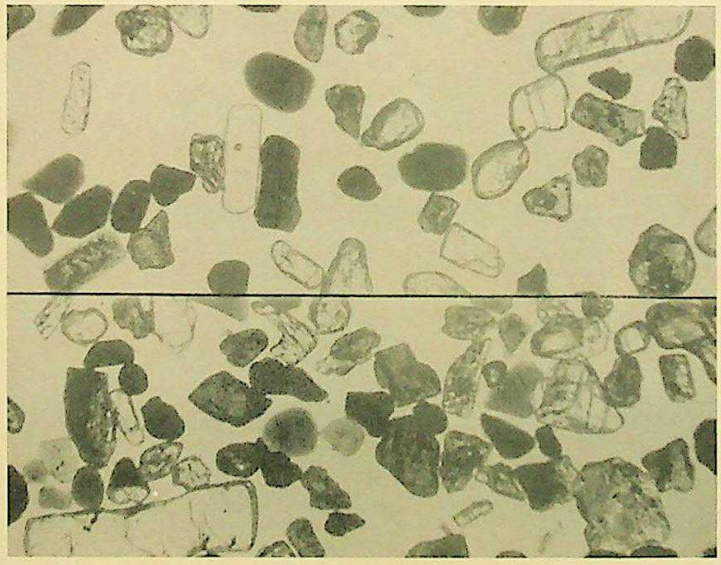

Figure 11.- The sample above is a dune sand from southern Alabama and consists of staurolite, kyanite, ilmenite, tourmaline, and rutile. The sample below is a canal sand from the same area. Kyanite, rutile, tourmaline, ilmenite, zircon, and staurolite are present in this slide. (Approximate mag., 26X.)

desired to obtain exactly balanced negatives, it is necessary to adjust the microscope accurately so that approximately the same amount of light is yielded by each tube.

The adjustment of the illumination in the two microscopic tubes can be made by means of a Lange ocular photocell. 12/ A photograph of this equipment and some of its properties is given in a paper by Faust and Cooke. 13/ Using this procedure, one of the fields on the stage of the comparison-microscope is covered with a black, opaque card and is shielded from reflected light by a black card set about the tube in cylindrical fashion on top of the stage. Even with these precautions, an exceedingly small amount of light appears in the tube from other sources. The ocular photocell is now placed on the ocular, and a deflection registered on a galvanometer in the ocular photocell circuit is noted. The black cards are now removed, and another measurement is made with the ocular photocell. The galvanometer deflection thus obtained represents the total illumination contributed by each tube. To equalize the illumination the light source may be shifted or the substage diaphragms may be closed slightly.

When the microscopic preparations are placed on the stage, the illumination is again upset. This is due to a number of factors, among which are unequal absorption of light by the contrasted materials; unequal concentration of particles per unit area; unequal sizes of the particles; unsymmetrical illumination within the field; touching of adjacent particles; and absorption of light by the mounting media. The elimination of these difficulties may be possible, as in figures 4, 5, 6, 7, and 11, or totally impossible, as in figure 9. Adjustments may be made by changing the fields under one of the microscope tubes by shifting the slide, the use of colored filters, shifting the mirror slightly, or closing one of the substage condensers to a small degree. With polarized light another difficulty is imposed by the isotropic and anistropic character of mineral grains, with consequent variation in the amount of birefringence. (See especially figs. 8 and 10.)

Even though it requires some care to produce good photomicrographs with the comparison-microscope, well-matched prints can be obtained. In normal photomicrographic work the preparation of well-matched prints is largely fortuitous.

Whereas, the matter of preparing photomicrographs is discussed here, the petrographer does not need them in his work with the polarizing comparison-microscope. They are, however, desirable for the illustration of reports.

12/ Lange, B., Photoelements and Their Applications. Part II: Reinhold Publishing Co., New York, 1938, chap. 1, p. 179.

13/ Faust, G. T., and Cooke, S. R. B., Ocular Photocell for the Rapid Determination of Projected Area of Opaque Particles: Bureau of Mines Rept. of Investigations 3460, 1939, 14 pp.

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